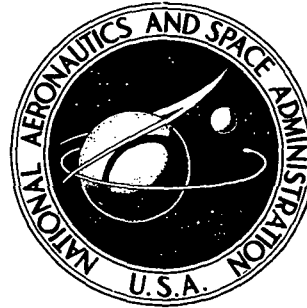


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BOUNDARY LUBRICATION OF FORMULATED C-ETHERS IN AIR TO 300° C

I - Phosphorus Ester Additives

by William R. Jones, Jr., and William F. Hady

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Cleveland, Ohio 44135

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BOUNDARY LUBRICATION OF FORMULATED C-ETHERS IN AIR TO 300° C

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SUMMARY

Friction and wear measurements were made on CVM M-50 steel lubricated with three C-ether (modified polyphenyl ether) formulations (phosphorus ester additives) in dry (<100 ppm H_2O) and wet air (RH 50 percent at 25° C (77° F)). Results were compared to those obtained with a fully formulated Type II ester and the C-ether base fluid. A ball-on-disk sliding friction apparatus was used. Experimental conditions were a 1-kilogram load (initial Hertz stress, 1×10^9 N/m²), a 17-meter-per-minute (100-rpm) disk surface speed, a 25° to 300° C (77° to 572° F) disk temperature range, and a 25-minute test duration.

The C-ether base fluid and the three C-ether formulations yielded lower wear than the Type II ester over the entire temperature range. In dry air (<100 ppm H_2O) the three C-ether formulations yielded lower wear than the C-ether base fluid from 100° to 300° C (212° to 572° F) but essentially the same wear from 25° to 100° C (77° to 212° F). In wet air (RH 50 percent at 25° C (77° F)) the three formulations yielded lower wear than the base fluid from 150° to 300° C (302° to 572° F) but higher wear from 25° to 150° C (77° to 302° F).

All C-ether fluids exhibited slightly higher friction coefficients than the Type II ester from 150° to 300° C (302° to 572° F). No differences were noted from 25° to 150° C (77° to 302° F).

In general, lower wear rates were observed with the C-ethers when tested in a wet air (RH 50 percent at 25° C (77° F)) as compared to a dry air (<100 ppm H_2O) atmosphere.

INTRODUCTION

Advanced aircraft and re-entry vehicles will place increased thermal stresses on hydraulic fluids and lubricants. Maximum fluid temperatures in excess of 316° C

(600° F) have been estimated for future applications (refs. 1 to 5). At these elevated temperatures, fluids must operate without appreciable degradation and must also provide effective lubrication for bearings and hydraulic system components.

Presently available fluids such as the super-refined mineral oils (refs. 6 and 7), hindered esters (refs. 3, 8, and 9), fluorinated polyethers (refs. 2, 3, and 10), and polyphenyl ethers (refs. 3, 7, 11, and 12) have one or more deficiencies which limit their use above 260° C (500° F). These deficiencies include poor oxidation stability, poor boundary lubricating characteristics, and corrosivity. In addition, because of the optimization of the high temperature properties, these fluids exhibit poor low temperature fluidity (high pour point).

The C-ethers, which are structurally related to the polyphenyl ethers, are a promising class of fluids for possible high temperature applications (refs. 13 and 14). They have excellent thermal stability (thermal decomposition temperature of 390° C (734° F) measured by isoteniscope), good oxidation stability to 260° C (500° F), and adequate pour points to -29° C (-20° F). They also exhibit low vapor pressure, high surface tension, and excellent shear stability. The main deficiencies of the C-ethers have been their poor boundary lubricating ability and poor wetting characteristics (refs. 7 and 15). Heat transfer (cooling) problems have also been encountered with this fluid class (ref. 2) and are probably a result of its poor wetting properties.

The objectives of this investigation were to (1) determine the friction and wear of CVM M-50 (consumable electrode vacuum melted) steel lubricated with three C-ether formulations (phosphorus ester additives) in dry (<100 ppm H₂O) and wet air (50 percent RH at 25° C (77° F)) from 25° to 300° C (77° to 572° F); and (2) to compare these results with those obtained with a fully formulated Type II ester (MIL-L-23699) and the C-ether base fluid.

Other experimental conditions included a 1-kilogram load (initial Hertz stress, 1×10^9 N/m²), a 17-meter-per-minute (100-rpm) surface speed, and a test duration of 25 minutes.

APPARATUS

The ball-on-disk sliding friction apparatus is shown in figure 1. The test specimens were contained inside a stainless steel chamber. The atmosphere was controlled with respect to moisture content. A stationary 0.476-centimeter-radius ball was placed in sliding contact with a rotating 6.3-centimeter-diameter disk. A sliding speed of 17 meters per minute (100 rpm) was maintained. A normal load of 1 kilogram (initial Hertz stress, 1×10^9 N/m²) was applied with a deadweight. Balls and disks were made of CVM M-50 tool steel. Disk and ball hardness was Rockwell C 62 to 64.

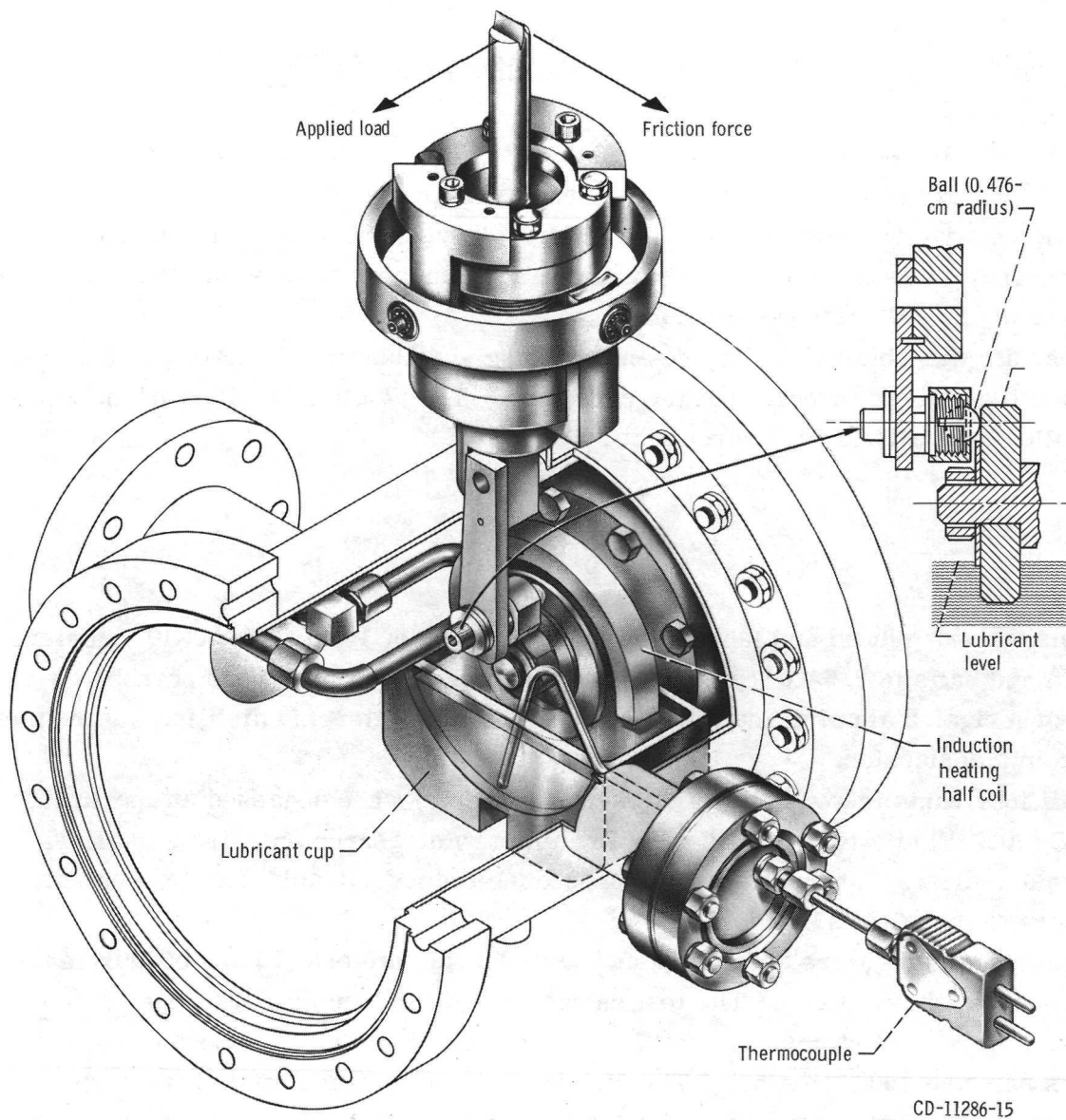


Figure 1, -Friction and wear apparatus.

The disk was partially submerged in a polyimide cup containing the test lubricant and was heated by induction. Bulk lubricant temperature was measured with a thermocouple. Disk temperature was monitored with an infrared pyrometer. Frictional force was measured with a strain gage and was recorded on a strip chart recorder.

MOISTURE MONITORING AND CONTROL

The two atmospheres used in this study were (1) wet air at a relative humidity of 50 ± 5 percent at 25°C (77°F) and (2) dry air (<100 ppm H_2O).

The relative humidity was monitored by a direct reading hygrometer accurate to ± 1.5 percent. The low water concentrations were monitored by a moisture analyzer with an accuracy of ± 10 parts per million.

Dry air was obtained by drying and filtering service air. Wet air was obtained by bubbling the dry gas through a water reservoir. The relative humidity of the wet air was controlled manually to 50 ± 5 percent at 25°C (77°F).

PROCEDURE

Disks were ground and lapped to a surface finish of 10×10^{-8} to 20×10^{-8} meters (4 to 8 $\mu\text{in.}$) and balls to 2.5×10^{-8} meters (1 $\mu\text{in.}$) rms. Specimens were scrubbed with a paste of levigated alumina and water, rinsed with tap water and distilled water, then placed in a desiccator.

All lubricants tested in dry air (except the ester) were degassed at approximately 150°C (302°F) at 2 torr for 1 hour. Preliminary measurements using the Karl Fischer technique indicate that this degassing procedure reduces dissolved water content in C-ethers to less than 20 ppm.

The specimens were assembled and 7×10^{-5} cubic meters (70 ml) of lubricant were placed in the lubricant cup. The test chamber (3.7×10^{-3} cu m or 3.7 liter vol) was purged with the test atmosphere for 10 minutes at a flow rate in excess of 5×10^{-2} cubic meters per hour (50 liters/hr). The disk was heated by induction to test temperature while rotating. The ball was then loaded against the disk. Test atmosphere flow rate was reduced to 3.5×10^{-2} cubic meters per hour (35 liters/hr), and a 6.9×10^{-3} -newton-per-square-meter (1-psig) pressure was maintained in the chamber. The lubricant was heated only by heat transfer from the rotating disk. The bulk lubricant temperature was essentially the same as the disk temperature at disk temperatures to 100°C (212°F). At disk temperatures of 200° and 300°C (392° and 572°F), the bulk oil temperatures stabilized at approximately 150° and 200°C (302° and 392°F), respectively.

Frictional force and bulk lubricant temperature were continuously recorded. Disk temperature was continuously monitored. Experiments were terminated after a 25-minute duration, and rider wear scar diameter was recorded.

EXPERIMENTAL LUBRICANTS

The experimental fluids used in these experiments were a formulated Type II ester, a C-ether base fluid, and three C-ether formulated fluids. Some typical properties of the test fluids appear in table I. Table II contains the additive contents of the test fluids.

TABLE I. - TYPICAL PROPERTIES OF THE EXPERIMENTAL FLUIDS

Properties ^a	C-ether base fluid	Type II ester
Kinematic viscosity, m ² /sec (cS)		
At 38° C (100° F)	2.5×10 ⁻⁵ (25)	2.8×10 ⁻⁵ (28)
At 99° C (210° F)	4.1×10 ⁻⁶ (4.1)	5.3×10 ⁻⁶ (5.3)
At 300° C (572° F)	6.9×10 ⁻⁷ (0.69)	^b 6.8×10 ⁻⁷ (0.68)
Pour point, °C (°F)	-29 (-20)	-60 (-75)
Flash point, °C (°F)	239 (445)	280 (535)
Fire point, °C (°F)	285 (540)	-----
Density at 38° C (100° F), kg/m ³ (g/ml)	1.19×10 ³ (1.19)	^c 0.990
Thermal decomposition (isoteniscope), °C (°F) ^d	390 (734)	316 (600)
Vapor pressure at 371° C (600° F), torr	140	-----
Surface tension at 23° C (73° F), N/cm (dynes/cm) ^d	44.8×10 ⁻⁴ (44.8)	-----
Erdco bearing rig deposit rating (Type II conditions) ^e	-----	26

^aManufacturer's data.

^bExtrapolated.

^cSpecific gravity (15.6° C/15.6° C (60° F/60° F)).

^dMeasured by authors.

^eBulk oil 227° C (440° F), oil in 204° C (400° F), bearing 260° C (500° F).

TABLE II. - ADDITIVE CONTENTS OF TEST FLUIDS

Formulated Type II ester	C-ether base fluid	C-ether formula- tion I	C-ether formula- tion II	C-ether formula- tion III
Antifoam, anticor- rosion, aromatic amine antioxi- dant, combined antioxidant and load carrying agent	Antifoam	Antifoam, straight chain aliphatic phosphorus ester	Antifoam, branched chain aliphatic phosphorus ester	Antifoam, branched chain aliphatic phosphorus ester, halogenated acid

Formulated Type II Ester

A fully formulated Type II ester was chosen as a reference fluid for these experiments. This lubricant is commercially available and meets General Electric D50TF1, Pratt and Whitney PWA 521B, and MIL-L-23699 lubricant specifications.

C-ether Base Fluid

The C-ether base fluid used in this study was originally reported in reference 13. This fluid is a blend of three-ring and four-ring components which are structurally similar to the polyphenyl ethers. This base fluid contains an antifoam additive.

C-ether Formulations (Phosphorus Ester Additives)

Formulation I. - The first C-ether formulation studied was the base fluid plus a straight chain aliphatic phosphorus ester. This additive should improve the boundary lubricating characteristics of the base fluid.

Formulation II. - The second C-ether formulation studied was the base fluid plus a branched chain aliphatic phosphorus ester. This is another additive to improve boundary friction and wear. This formulation has been previously studied in reference 2. It performed satisfactorily in short-term (3-hr) bearing tests at a bearing temperature of 316° C (600° F) in air and nitrogen. However, it required higher oil flow rates to stabilize the bearing temperature than did ester and synthetic paraffinic hydrocarbon lubricants.

Formulation III. - The third C-ether formulation studied is identical to formulation II except that a halogenated acid has been added to improve fluid wettability. This particular formulation has also been studied previously (ref. 16). This fluid provided marginally adequate boundary lubrication in small scale bearing screening tests at 316° C (600° F) for 100 hours in nitrogen.

RESULTS AND DISCUSSION

Wear

Formulated Type II ester. - This fluid was chosen as a reference fluid because it appeared to be a typical example of the polyol ester group of MIL-L-23699 lubricants.

Wear results for this fluid appear in figure 2. No significant differences in wear were observed between the dry and wet air results. Therefore, a single wear-temperature curve appears in figure 2.

The wear rate is essentially constant at 1.4×10^{-13} cubic meters per minute over the entire temperature range. Two typical wear scars obtained with this ester at disk temperatures of 50° and 150° C (122° and 302° F) in dry air appear in figures 3(a) and (b). Metallic wear debris can be seen in the contact exit. This debris is shown enlarged in figures 3(c) and (d). The level of wear and the appearance of the wear scar indicates only marginally adequate lubrication was obtained with this ester.

C-ether base fluid. - Wear results for this base fluid appear in figure 4. At 25° C (77° F) in dry air the wear rate was about 7×10^{-14} cubic meters per minute. The wear rate decreased to a minimum of approximately 4×10^{-14} cubic meters per minute at 100° C (212° F) and then gradually increased to 10^{-13} cubic meters per minute at 300° C

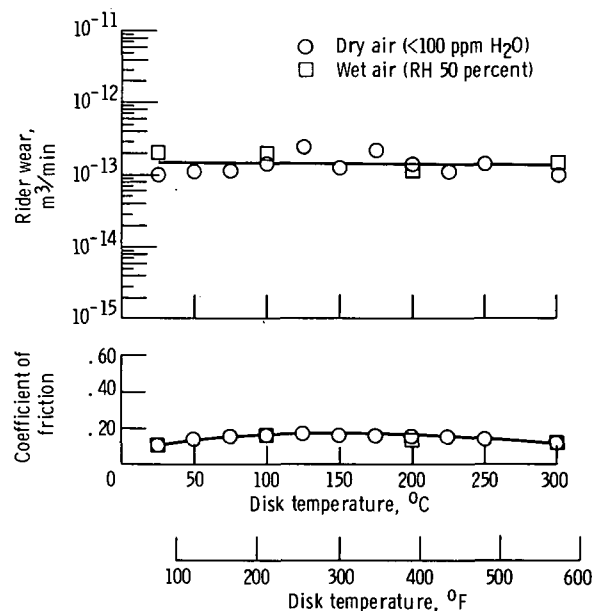
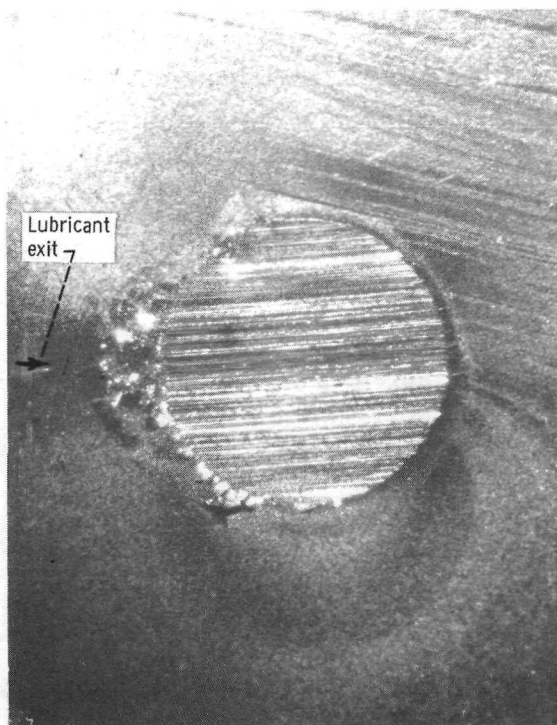
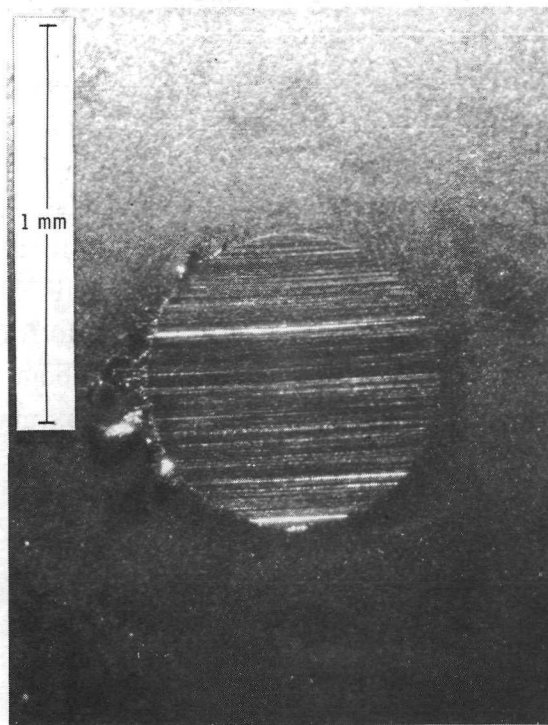


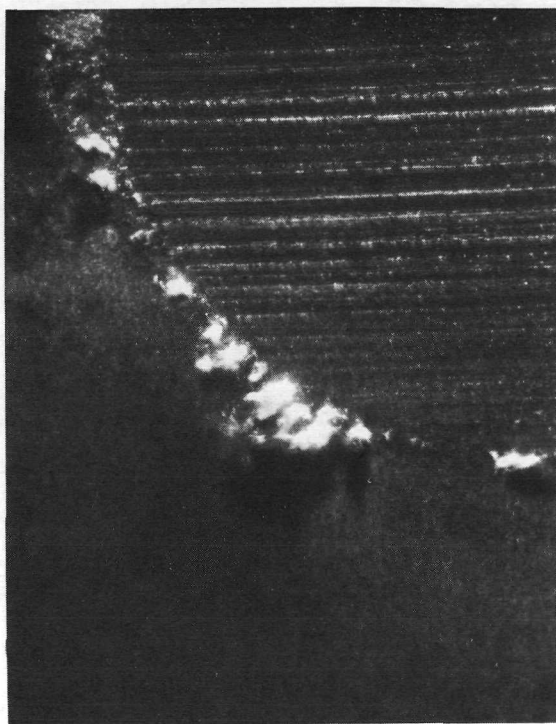
Figure 2. - Coefficient of friction and rider wear as a function of disk temperature for a fully formulated type II ester. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, 100-rpm disk speed, dry air (<100 ppm H_2O) and wet air (RH 50 percent), and 25-minute test duration.



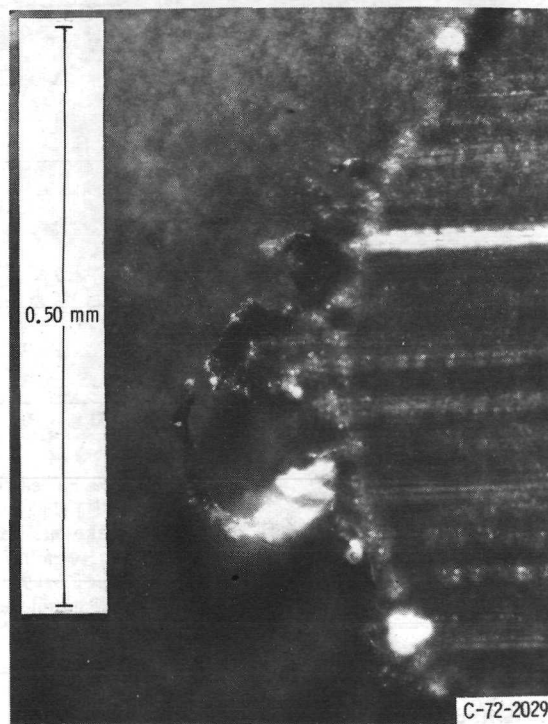
(a) 50° C (122° F).



(b) 150° C (302° F).



(c) Enlarged portion of (a).



(d) Enlarged portion of (b).

Figure 3. - Typical rider wear scars for a formulated Type II ester. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, dry air (<100 ppm H₂O), M-50 steel test specimens, and 25-minute test duration.

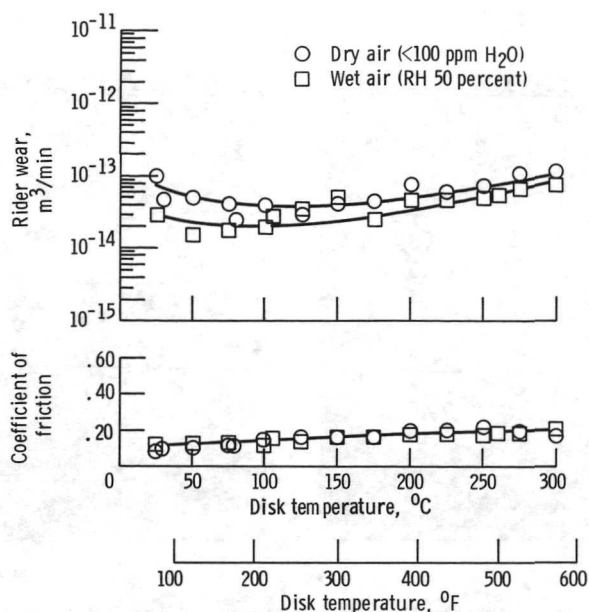


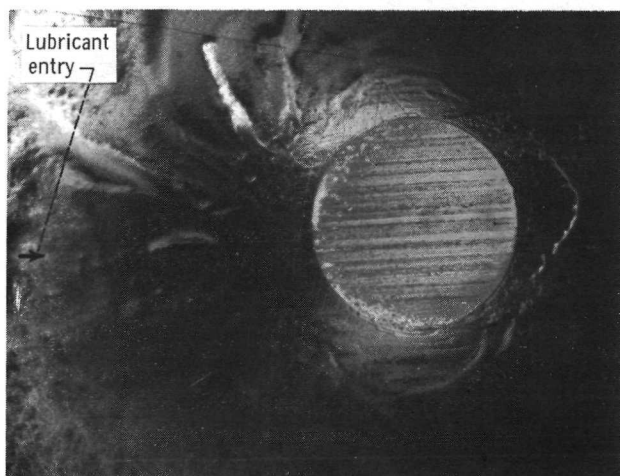
Figure 4. - Coefficient of friction and rider wear as a function of temperature for a C-ether base fluid. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, 100-rpm disk speed, dry air (<100 ppm H₂O) and wet air (RH 50 percent), and 25-minute test duration.

(572° F). In wet air a similar wear-temperature curve is observed but with lower wear rates over the entire temperature range. The effect of moisture in reducing wear was more pronounced at the lower temperatures. Two typical wear scars for the C-ether base fluid in dry air at disk temperatures of 50° and 225° C (122° and 437° F) appear in figure 5. Lubricant reaction products are noted in the inlet region to the contact.

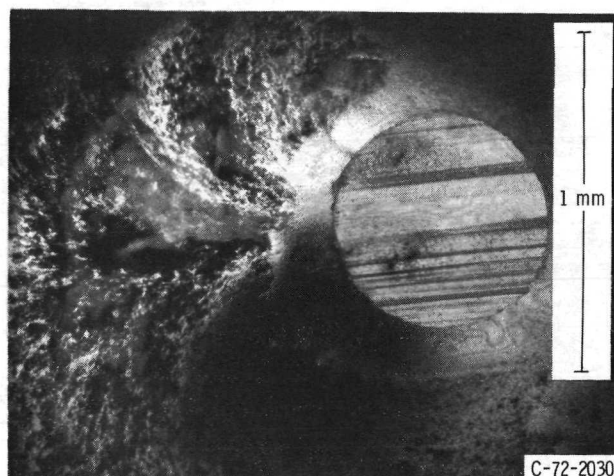
A comparison between the friction and wear results for the Type II ester and the C-ether base fluid appears in figure 6. In both dry and wet air the C-ether base fluid yielded lower wear than the Type II ester over the entire temperature range. The maximum difference in wear occurred at about 100° C (212° F). At 100° C (212° F) the ester exhibited a wear rate approximately seven times greater in wet air and four times greater in dry air than the wear rate observed for the C-ether base fluid.

C-ether formulations. - Wear results for C-ether formulations I, II, and III appear in figure 7. No significant differences in wear were observed between the dry and wet air results for formulations I and II. Therefore, only a single wear-temperature curve appears in figures 7(a) and (b). Formulation III, however, yielded lower wear rates in wet air than in dry air from 100° to 300° C (212° to 572° F).

A qualitative comparison of the wear results for the three formulations and the base fluid appears in figure 8. The dry air results of figure 8(a) indicate essentially no difference in wear rate for the three formulations and the base fluid from 25° to 100° C



(a) 50° C (122° F).



(b) 225° C (437° F).

Figure 5. - Typical rider wear scars for a C-ether base fluid. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, dry air (<100 ppm H₂O), M-50 steel test specimens, and 25-minute test duration.

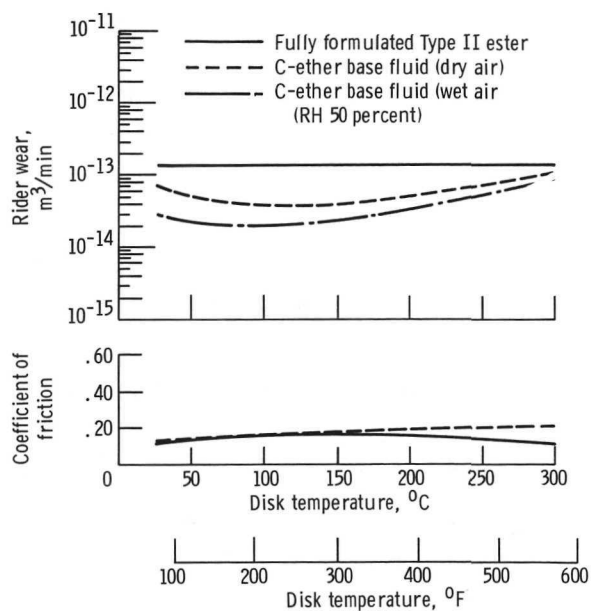


Figure 6. - Comparison of friction and wear results for a formulated Type II ester and a C-ether base fluid from 25° to 300° C (77° to 572° F). Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, 100-rpm disk speed, dry air (<100 ppm H₂O) and wet air (RH 50 percent), and 25-minute test duration.

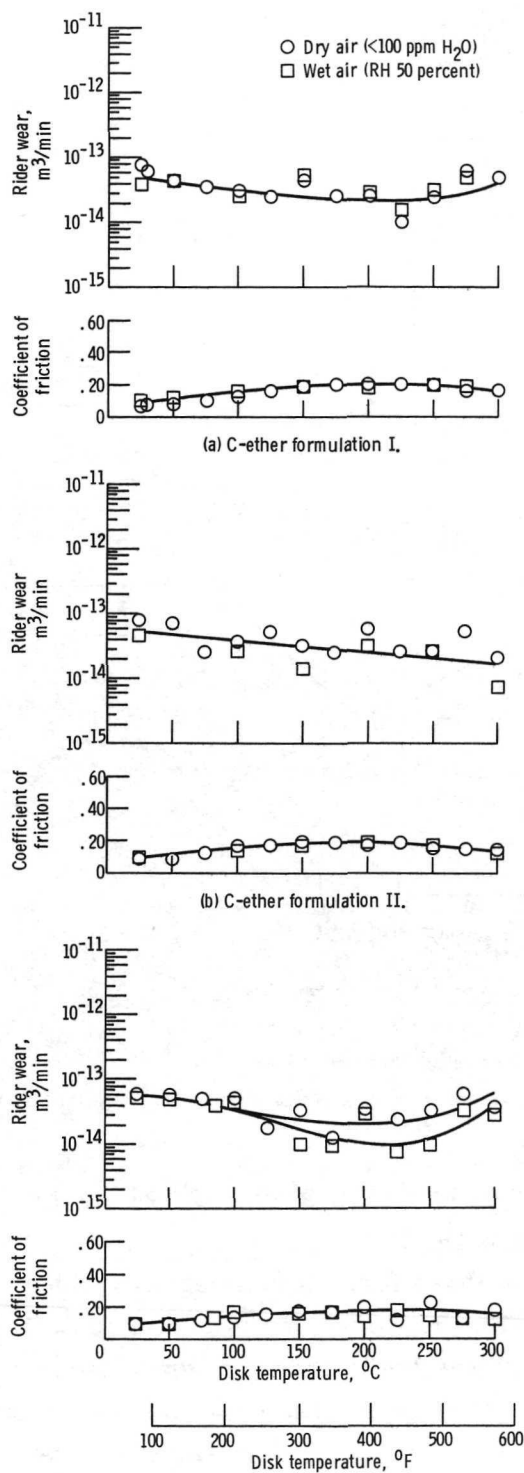


Figure 7. - Coefficient of friction and rider wear as a function of temperature for three C-ether formulations. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, 100-rpm disk speed, dry air (<100 ppm H₂O) and wet air (RH 50 percent), and 25-minute test duration.

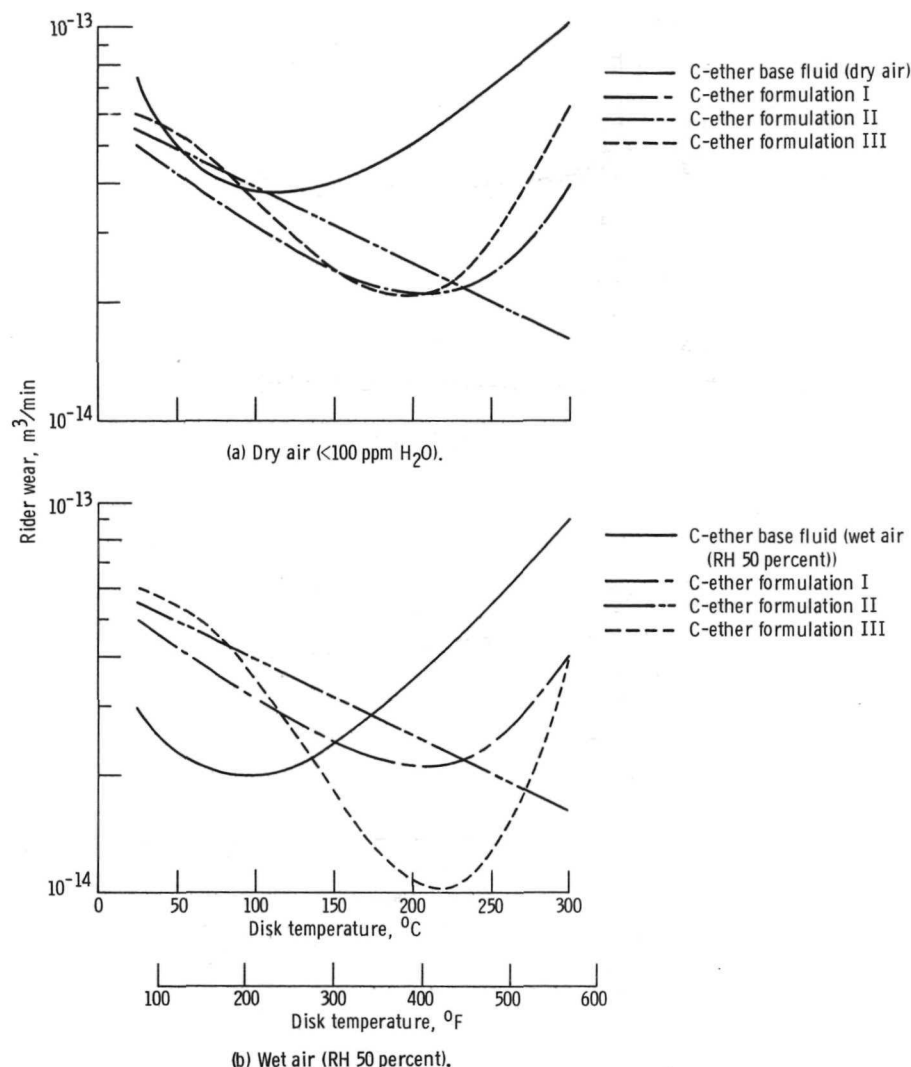


Figure 8. - Rider wear as a function of temperature for a C-ether base fluid and three C-ether formulations.

(77° to 212° F). From 100° to 300° C (212° to 572° F) all three formulations yielded lower wear rates than the base fluid.

In wet air (fig. 8(b)), the three formulations again yielded lower wear rates above approximately 150° C (302° F). However, from 25° to 150° C (77° to 302° F) the wear rate of the C-ether base fluid was lower than the wear rates of the three formulations. The increasing effectiveness of the additives with increasing temperature is most likely related to their greater reactivity at the higher temperatures.

Coefficient of Friction

Figure 6 contains a comparison between the friction coefficients for the Type II

ester and the C-ether base fluid. Figure 7 shows the friction-temperature curves for each of the three formulations. As shown in these figures, all C-ether fluids exhibited slightly higher friction coefficients than the Type II ester from 150° to 300° C (302° to 572° F). Similar friction coefficients for all fluids were observed from 25° to 150° C (77° to 302° F). Above 200° C (392° F) the three C-ether formulations yielded slightly lower friction coefficients than the C-ether base fluid.

Effect of Moisture on Friction and Wear

Lower wear rates (40 to 80 percent of the dry air wear rate) were observed with the C-ether base fluid when tested in wet air as compared to dry air (fig. 4). This is not surprising since aromatic fluids usually have lower wear rates in the presence of moisture (ref. 17). In previous work, similar reductions in wear were observed with a five-ring polyphenyl ether in wet air from 150° to 350° C (302° to 662° F) (ref. 12).

Testing in a wet as opposed to a dry air atmosphere had little effect on the wear rates for formulations I and II as shown in figure 7. However, lower wear rates were observed with formulation III from 125° to 300° C (257° to 572° F) in wet air. The amount of moisture in the test atmosphere had no significant effect on the friction coefficients for any of the test fluids.

TABLE III. - SUMMARY OF FRICTION AND WEAR RESULTS

Disk temperature		Test fluids				
		Type II ester	C-ether base fluid	C-ether formulation I	C-ether formulation II	C-ether formulation III
°C	°F					
Coefficient of friction						
25	77	0.10	0.12	0.10	0.10	0.09
100	212	.16	.16	.16	.15	.14
200	392	.16	.18	.20	.18	.18
300	572	.12	.20	.16	.12	.16
Rider wear, m ³ /min						
25	77	14×10 ⁻¹⁴	7.5×10 ⁻¹⁴	5.0×10 ⁻¹⁴	5.5×10 ⁻¹⁴	6.0×10 ⁻¹⁴
100	212	14	^a 3.0	3.1	3.9	3.6
200	392	14	^a 2.0	2.1	2.5	^a 3.5
300	572	14	^a 3.5	4.0	1.6	^a 1.1
			^a 9.0			^a 4.0

^aWet air (RH 50 percent) results.

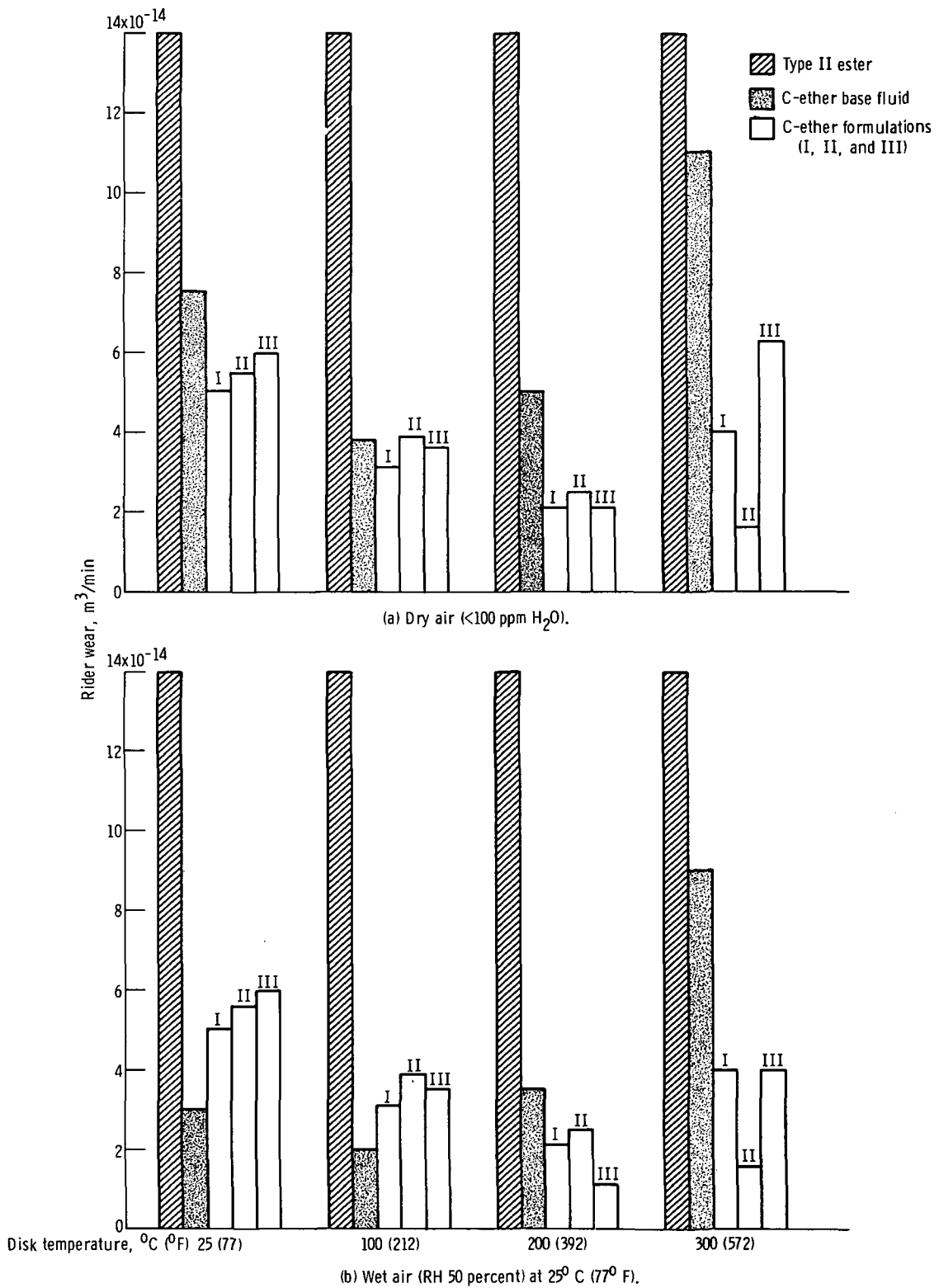


Figure 9. - Rider wear at four disk temperatures for type II ester, C-ether base fluid, and three C-ether formulations. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, 100-rpm disk speed, and 25-minute test duration.

A summary of friction and wear results for all test fluids at four selected temperatures of 25°, 100°, 200°, and 300° C (77°, 212°, 392°, and 572° F) appears in table III. Figure 9 also summarizes the wear rates for all fluids at the aforementioned selected temperatures in both dry and wet air.

It must be pointed out that the test conditions of this study are only a part of the spectrum of conditions that a lubricant or hydraulic fluid would be subjected to in service. Quite different results may be obtained under higher speeds, higher loads, and different metallurgies.

SUMMARY OF RESULTS

The friction and wear of CVM M-50 steel lubricated with three C-ether formulations (phosphorus ester additives) in dry (<100 ppm H₂O) and wet air (RH 50 percent at 25° C (77° F)) were determined using a ball-on-disk sliding friction apparatus. Disk temperature range was 25° to 300° C (77° to 572° F). Other conditions were a 1-kilogram load (initial Hertz stress, 1×10^9 N/sq m), a 17-meter-per-minute (100 rpm) sliding speed, and a 25-minute test duration. Results were compared to those obtained with a formulated Type II ester and the C-ether base fluid. The major results were the following:

1. The three C-ether formulations and the C-ether base fluid yielded lower wear than the Type II ester over the entire temperature range in both wet and dry air.
2. In dry air the three C-ether formulations yielded lower wear than the C-ether base fluid from 100° to 300° C (212° to 572° F) but essentially the same wear from 25° to 100° C (77° to 212° F). In wet air the three formulations yielded lower wear than the base fluid from 150° to 300° C (302° to 572° F) but higher wear from 25° to 150° C (77° to 302° F).
3. All C-ether fluids exhibited slightly higher coefficients of friction than the Type II ester from 150° to 300° C (302° to 572° F). No differences were noted from 25° to 150° C (77° to 302° F).
4. In general, lower wear rates were observed with the C-ethers when tested in a wet air (RH 50 percent at 25° C (77° F)) as compared to a dry air (<100 ppm H₂O) atmosphere.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 24, 1972,
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